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TO: Assistant Commissioner for Patents  
Box Patent Applications  
Washington D.C. 20231

Attorney Docket No.076397/0124

(must include alphanumeric codes if no inventors named)

**UTILITY PATENT APPLICATION TRANSMITTAL**  
(new nonprovisional applications under 37 CFR 1.53(b))

Transmitted herewith for filing is the patent application of:

INVENTOR(S): Klaus BUECHER, Ulrich MEYER-BLUMENROTH, Klaus NOLL and  
Todd REUS

TITLE: MEMBRANE ELEMENT AND PROCESS FOR ITS PRODUCTION

In connection with this application, the following are enclosed:

**APPLICATION ELEMENTS:**

XX Specification - 12 TOTAL PAGES

(preferred arrangement:)

- Descriptive Title of the Invention
- Cross Reference to Related Applications
- Statement Regard Fed sponsored R&D
- Reference to Microfiche Appendix
- Background of the Invention
- Brief Summary of the Invention
- Brief Description of the Drawings (if filed)
- Detailed Description
- Claim(s)
- Abstract of the Disclosure

XX Drawings - Total Sheets 6

XX Declaration and Power of Attorney - Total Sheets 3

XX Newly executed (original or copy)

     Copy from a prior application (37 CFR 1.63(d))

(relates to continuation/divisional boxes completed) - NOTE: Box below

     DELETION OF INVENTOR(S) - Signed statement attached deleting inventor(s)  
named in the prior application, see 37 CFR 1.63(d) (2) and 1.33(b).

     Incorporation By Reference (useable if copy of prior application  
Declaration being submitted)

The entire disclosure of the prior application, from which a COPY of the  
oath or declaration is supplied as noted above, is considered as being  
part of the disclosure of the accompanying application and is hereby  
incorporated by reference therein.

     Microfiche Computer Program (Appendix)

     Nucleotide and/or Amino Acid Sequence Submission (if applicable,  
all necessary)

     Computer Readable Copy

     Paper Copy (identical to computer copy)

     Statement verifying identify of above copies

**ACCOMPANYING APPLICATION PARTS**

XX Assignment Papers (cover sheet & document(s))

     37 CFR 3.73(b) Statement (when there is an assignee)

     English Translation Document (if applicable)

☒ Information Disclosure Statement (IDS) with PTO-1449. 1 Copy of IDS Citation  
☒ Preliminary Amendment  
☒ Return Receipt Postcard (MPEP 503)  
☐ Small Entity Statement(s)  
☐ Statement file in prior application, status still proper and desired.  
☒ Certified Copy of Priority Document(s) with Claim of Priority  
(if foreign priority is claimed).  
☒ OTHER: Check for \$800.00

If a **CONTINUING APPLICATION**, check appropriate box and supply the requisite information:

☐ Continuation ☐ Divisional ☐ Continuation-in-part (CIP)  
of prior application Serial No.       .

☐ Amend the specification by inserting before the first line the following sentence: --This application is a        continuation,        divisional or        continuation-in-part of application Serial No.       , filed       .--

**CORRESPONDENCE ADDRESS:**

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**FEE CALCULATIONS:** (Small entity fees indicated in parentheses.)


(1) For	(2) Number Filed	(3) Number Extra	(4) Rate	(5) Basic Fee \$760 (\$380)
Total Claims	14 - 20 =	0	x \$18 (x \$ 9)	0.00
Independent Claims	2 - 3 =	0	x \$78 (x \$39)	0.00
Multiple Dependent Claims			\$260 (\$130)	0.00
Assignment Recording Fee per property			\$40	40.00
TOTAL FEE:				\$800.00

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A check in the amount of the above TOTAL FEE is attached. If payment is enclosed, this amount is believed to be correct; however, the Commissioner is hereby authorized to charge any deficiency or credit any overpayment to Deposit Account No. 19-0741.

Respectfully submitted,

Date: June 10, 1999  
Docket No.: 076397/0124

  
Richard L. Schwaab  
Reg. No. 25,479

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Attorney Docket No. 076397/0124

In re patent application of

Klaus BUECHER et al.

Serial No.: Unassigned

Filed: June 10, 1999

For: MEMBRANE ELEMENT AND PROCESS FOR ITS PRODUCTION

PRELIMINARY AMENDMENT

Assistant Commissioner for Patents  
Washington, D.C. 20231

Sir:

Prior to examination of the above-identified application, Applicants respectfully request that the following amendments be entered into the application:

IN THE CLAIMS:

Please amend the following claims:

Claim 3, line 1, delete "or 2".

Claim 4, line 1, delete ", 2 or 3".

Claim 11, line 1, delete "or 9".

Claim 12, line 1, delete "or 9".


Attorney Docket No.: 76397/124

REMARKS

Applicants respectfully request that the foregoing amendments to Claims 3, 4, 11 and 12 be entered in order to avoid this application incurring a surcharge for the presence of one or more multiple dependent claims.

Respectfully submitted,

June 10, 1999

  
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## Membrane element and process for its production

The invention relates to novel membrane elements, a process for their production and their use as filters or  
5 separators, for example for ultrafiltration, nanofiltration, reverse osmosis, gas separation or pervaporation.

For the purposes of the invention, membrane elements are  
10 devices in which the membranes which carry out the actual function of separation or retention - and which are usually very fragile - have been arranged in such a way as to withstand operating conditions which are frequently severe.

15 Membrane elements may have various structures. Those known as spirally wound elements are widely used. They are composed of one or more doubled layers of membrane 1 with their active separating layers facing outward. These  
20 double layers are in each case bonded or fused to one another on three sides, forming what are known as membrane pockets. The open side is bonded to the permeate pipe 4, which has perforations or holes 7 in the region of the membrane layers. Once this has been done the inner  
25 sides of the membrane pockets are then in communication only with the permeate pipe 4. The membrane pockets are wound around the permeate pipe. A feed 2 enters at the end side of the element and passes through it axially between the membrane pockets. A suitable spacer 6 ensures  
30 good cross-flow and the best possible mixing of the feed stream at the membrane surface. A pressure difference causes permeate 5 to enter the membrane pockets from both sides. A drainage layer composed of a specific permeate spacer 3 assures good outflow to the permeate pipe, the  
35 holes 7 in which provide for outflow (see Fig. 1).

Depending on the application, the semipermeable membranes used may be micro-, ultra- or nanofiltration membranes or reverse osmosis membranes, gas separation membranes or pervaporation membranes.

5

Elements of this type are used in waste-water treatment, in the food and drink industry, in the pharmaceutical industry, in the production of drinking water, in the separation of gas mixtures, etc. A description of a typical structure of an element is found in R.E. Larson, et al. "Test results on FT-30 eight-inch-diameter seawater and brackish water reverse osmosis element", Desalination 46 (1983) pp. 81/90. Another form of construction is that known as "pleated elements". In this  
10 the membranes are laid in folds and likewise are arranged around a permeate-collecting pipe. Elements of this structure are, for example, commercially available from Daicen, Japan. Examples: MOLSEP PV04-GN-DUY-L000 and MOLSEP PV08-VV-DUY-L000.

15  
20

When elements of this type are in operation they have mostly been built into casings, also termed pressure pipes. In the case of cross-flow filtration, very large amounts of fluid frequently flow across the membrane  
25 elements in the pressure pipes. To prevent the elements deforming during operation, their ends are usually provided with what are known as anti-telescoping devices (ATD) 8 and surrounded by suitable sheaths which improve their stability. The ATDs may be firmly bonded to the  
30 membrane elements, or placed over their ends, or attached to the inside of the pressure pipe. The sheaths are frequently composed of the material also used as spacer in the membrane element, for example extruded polypropylene baskets, windings with adhesive tape (made  
35 from PVC, polypropylene or polyester) or hard shells made from glass-fibre-reinforced plastic (GFP). However, under

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extreme operating conditions these sheaths can frequently become unable to fulfill their functions, and are damaged or deform. For example, most sheaths produced from adhesive tapes lose their stability at temperatures above 5 50°C. Extruded polypropylene baskets also retain only very limited dimensional stability at elevated temperatures. Membrane elements with hard GRP shells can be attacked by relatively concentrated acids or alkalis, or by solvent contents in the feed stream. This may 10 become noticeable, for example, through a fall in strength or through flaking of the surface. Even when operating at low levels of cross-flow (some plants use what is known as dead-end operation, with no cross-flow) the operating conditions can place the membrane elements 15 under stress, e.g. during rinsing or cleaning or as a result of unintended operating conditions (severe change in the feed stream, high temperatures, sudden changes in pressure, various service failures, etc.).

20 It is also vital, for use of membrane elements in gas separation and pervaporation, to avoid deformations. Agressive components in the feed stream may likewise impair the integrity of the membrane elements here, such as extreme or undesired operating conditions 25 (considerable change in the feed stream, high temperatures, pressure shocks, general operating errors, etc.).

The object of the present invention was therefore to 30 provide membrane elements which do not have the disadvantages described above and which in particular have sheaths which are more stable than those of the prior art. Ideally, the membrane elements should combine the following properties:

35

- reliable operation in contact with relatively

concentrated acids and alkalis, even when combined with high temperatures (up to 100°C)

- no detachment of parts of the sheath on contact with solvents

5       • very high mechanical stability, comparable with that of hard GRP shells, but without the disadvantages of these

- sheath made from a material which is also already used in the actual membrane element.

10

This object is achieved by a membrane element comprising a core and a sheath which encapsulates the core, where the sheath is formed from polymer films which overlap one another at least to some extent and have been fused to  
15 one another in the area of the overlap.

The object is also achieved by a process for producing membrane elements, in which a membrane core is provided with a sheath by winding a functionalized polymer film  
20 around the membrane core, where individual layers of the polymer film overlap one another, at least in some areas, and energy is supplied to fuse the polymer films to one another at least in these areas.

25 The novel membrane elements do not have the disadvantages of conventional sheaths. By selecting a suitable polymer film for the sheath, reliable operation may be achieved in contact with relatively concentrated acids and alkalis, even when combined with high temperatures (up to  
30 100°C). In addition, it is virtually impossible for parts of the sheath to detach on contact with solvents. The sheaths produced by the process described below have very high mechanical stability, comparable with that of hard GRP shells, but without the disadvantages of these. In  
35 most cases it is possible to produce the sheath from a material which is also already used in the actual



membrane element.

The polymer film web for the sheath is composed of one or more layers of a polymer film with at least one functionalized surface. More than one layer of polymer films may be built up using films of identical or different types.

Particularly suitable polymer films for the application described here are those made from polypropylene (filled, unfilled, with microvoids, or filled and with microvoids). Other suitable films are based on polyester, in particular polyethylene terephthalate (filled, unfilled, with microvoids, or filled and with microvoids). Films made from PVC are also suitable, even though their chemical/thermal stability is lower.

For the purposes of the present invention, "functionalized" means that the nature of the films is such that when energy is supplied they can fuse to one another, and "fuse" means that the materials of the two films brought into contact and supplied with energy intermix in the area of contact virtually without any discernible phase boundary, this mixing taking place only at elevated temperatures, i.e. above about 70°C, preferably above about 100°C. This may be achieved, for example, by applying a sealing layer to at least one surface, the sealing layer having a lower melting temperature than the base film. For polypropylene films this may, for example, be a C<sub>2</sub>/C<sub>3</sub>-, C<sub>2</sub>/C<sub>4</sub>-, C<sub>3</sub>/C<sub>4</sub>- and/or C<sub>2</sub>/C<sub>3</sub>/C<sub>4</sub>-copolymer outer layer. Use may generally be made of any (thermoplastic) film in which a suitable process, e.g. coextrusion, has been used to provide at least one functionalized surface (e.g. sealing layer with a lower melting point than the base film).

The core of the membrane element may be constructed in various ways. According to the invention, preference is given to what are known as spirally wound elements. They are composed of one or more doubled layers of membrane  
5 with their active separating layers facing outward. These double layers are in each case bonded or fused to one another on three sides, and form what are known as membrane pockets. The open side is bonded to the permeate pipe, which has perforations or holes in the region of  
10 the membrane layers. The membrane pockets are then wound around the permeate pipe. It is useful to wind a spacer, such as a sheet of polypropylene net, together with the membrane pockets around the permeate pipe. The end sides of the resultant rolls are then provided with anti-  
15 telescoping devices which prevent telescoping of the wound-on membrane elements.

The sheath according to the invention may be applied to the core in various ways. A polymer film web 10 which has  
20 at least one functionalized surface is wound spirally around the cylindrical membrane element 9 (see Fig. 2).

The individual windings 11a here may overlap one another fully or to some extent (Fig. 3), or lie flush and  
25 alongside one another 11b (Fig. 4), or be applied at a distance from one another 11c Fig. 5). It is useful to apply more than one layer of these windings: from 1 to 400 layers, depending on the degree of overlapping and the thickness of the polymer film web. The thickness of  
30 the sheath is from 0.3 to 28 mm.

When the windings are applied, inclusion of air between the individual layers should be avoided. A suitable web tension needs to be set for this purpose. A pinch roll 12  
35 may optionally be used (Fig. 6), in which case the force which is applied should be adjusted so that air

inclusions are reliably prevented at the web tension used. The web tension may be adjusted within the range from 1N to 500N for web widths up to 100 mm and up to 1000 N for a web width of about 2000 mm. Wider web widths  
5 may also be used in principle if required by the length of the membrane element. However, the maximum web width should be selected with regard to the length of the membrane element.

10 Depending on width and thickness, the windings of the polymer film web are applied at a speed of from 0.1 to 300 m/min.

The bond between the individual layers of the polymer  
15 film web is produced by supplying energy, preferably by suitable heat treatment.

If the heat treatment takes place during the winding-on process, it should be carried out in such a way that the  
20 functionalized surfaces produce a bond between the individual layers, and the two surfaces are preferably fused to one another in the overlap area. The polymer film web or the sheath is heated, for example, by hot air, flame, infrared radiation, microwaves, one or more  
25 hot pinch rolls, or any other suitable heat source.

During winding around the core, the polymer film web is heated in such a way as to produce the bond (fusion) immediately when the individual layers come into contact,  
30 or is heated to the extent that a small amount of further heating of the sheath causes bonding (fusion) of the individual layers. The heating process should be carried out in such a way as to avoid significant change in stability or appearance.

35

An alternate method of bringing about the bond between

the individual layers during the winding-on process is ultrasound welding.

Another way of producing the bond is firstly to apply all  
5 of the layers of the winding and to use the heat treatment on the membrane element surrounded by the winding. Here, again, the heating may be by hot air, flame, infrared radiation, microwaves or any other suitable heat source. The heating process should be  
10 carried out in such a way as to produce a bond between all of the layers of the sheath, via their functionalized surfaces. The temperature and the exposure duration should be adjusted so that even the inner layers of the polymer film web become bonded to one another while  
15 avoiding any significant change in the stability or appearance of the outer layers which under some circumstances may be hotter (e.g. in the case of hot-air heating). This can be determined using simple experiments.

20 The abovementioned processes may also be combined, so that different parts of the sheath are produced by different processes. For example, a thin sheath may be applied using the last process mentioned, and this may be  
25 reinforced in a second step using the first process mentioned. The sequence may also be reversed, or one process may be replaced by the second process mentioned. The thicknesses of the sheaths produced by the respective processes may be adjusted within the range from 0.3 to  
30 28 mm.

In the abovementioned processes windings may also be produced not only by using films with one or more layers but also by simultaneous winding-on of films with one or  
35 more layers. It is entirely possible here for the films to be different from one another.

The same applies to stepwise winding, where a first polymer film is used in a first step and other films are also used in one or more subsequent steps.

- 5 However the process is carried out, it must be ensured that the membrane of the membrane element does not become damaged anywhere on the active surface.

What is claimed is

1. A membrane element comprising a core (9) and a sheath which encapsulates the core, where the sheath is formed from polymer films (10) which overlap one another at least to some extent and have been fused to one another in the area of the overlap.
2. A membrane element as claimed in claim 1, wherein the polymer film (10) is a polypropylene film or polyester film.
3. A membrane element as claimed in claim 1 or 2, wherein the polymer film (10) has at least one functionalized surface.
4. A membrane element as claimed in claim 1, 2 or 3, wherein the polymer film (10) is a coextruded film.
5. A membrane element as claimed in claim 4, wherein the coextruded film (10) is composed of a base layer and of at least one outer layer, where the melting point of the outer layer polymer is lower than that of the base layer polymer.
6. A membrane element as claimed in claim 5, wherein the melting point of the outer layer polymer is from 70 to 130°C.
7. A process for producing a membrane element, in which a membrane core (9) is provided with a sheath by winding a functionalized polymer film (10) around the membrane core (9), where individual layers (11) of the polymer film (10) overlap one another, at least in some areas, and energy is supplied to fuse

the polymer films to one another at least in these areas.

- 5        8.    The process as claimed in claim 7, wherein the polymer film (10) is wound as a layer spirally around the membrane core (9), where the individual laps of the layer (11a) overlap to some extent.
- 10       9.    The process as claimed in claim 7, wherein the polymer film (10) is wound as a layer spirally around the membrane core (9), where the individual laps of the layer (11b) are laid alongside one another, without overlapping each other, and form a first layer, and where at least one further layer of
- 15       polymer film (10) layers lying alongside one another is wound over the first layer, and this is then fused to the layer lying thereunder.
- 20       10.   The process as claimed in claim 7, wherein the thickness of the sheath is from 0.3 to 28 mm.
- 25       11.   The process as claimed in claim 8 or 9, wherein the width of the layer (10) is from 10 to 100 mm.
- 30       12.   The process as claimed in claim 8 or 9, wherein the width of the layer (10) is from 100 to 2000 mm.
- 35       13.   The process as claimed in claim 11, wherein the layer (10) is wound with a web tension of from 1 to 500N.
14.    The process as claimed in claim 12, wherein the layer (10) is wound with a web tension of from 100 to 1000N.

## Abstract

**Membrane element and process for its production**

The invention relates to novel membrane elements, a process for their production and their use as filters, for example for ultrafiltration, nanofiltration, reverse osmosis, gas separation or pervaporation. The invention relates in particular to a membrane element comprising a core 9 and a sheath which encapsulates the core, where the sheath is formed from polymer films 10 which overlap one another at least to some extent and have been fused to one another in the overlap area.



TEST RESULTS ON FT-30 EIGHT-INCH-DIAMETER  
SEAWATER AND BRACKISH WATER REVERSE OSMOSIS ELEMENTS

By R.E. Larson, R.J. Petersen, and P.K. Eriksson  
FilmTec Corporation, 7200 Ohms Lane, Minneapolis, MN 55435 USA

ABSTRACT

Spiral-wound reverse osmosis elements formed of FT-30 membrane have been used for a variety of desalting applications around the world. The membrane was originally developed to satisfy the requirement for a true single-pass seawater membrane that would maintain its salt rejection characteristics while operating in a variety of seawater feeds. Later test results proved its applicability as a brackish-water desalting membrane as well, and FT-30 elements are now operating over a broad spectrum of feedwaters, ranging from tapwater to difficult industrial separations.

The FT-30 membrane is noncellulosic and is of a thin-film composite configuration. It satisfies the main requirement of having high flux and excellent salt rejection, but in addition it is chemically stable, can operate over a broad range of pH, is partially resistant to chlorine exposure and shows good stability at high feedwater temperatures. These latter characteristics facilitate both acid and caustic cleaning, an important consideration under fouling conditions.

Considerable field application experience has been obtained with FT-30 membrane elements over the past three years. This experience has given excellent results, although it has been limited to small elements — the largest manufactured by FilmTec until recently was 4 X 21 inches and gave 600 gpd on seawater and 1200 gpd on brackish water. In early 1982 4- X 40-inch elements began to be produced and used commercially. Then in the summer of 1982, 8- X 40-inch elements were manufactured for the first time, meeting the design goals of single-pass seawater performance with product flows of over 5,000 gpd, and under brackish water conditions with flows of over 10,000 gpd. These large elements are now being tested on seawater and brackish water feeds in the MidEast, in England, in Florida, and in several other locations. Single-pass seawater performance data and brackish-water test results are presented. Eight-inch elements are now being produced in commercial quantities for use in large reverse osmosis installations.

RESULTATS DE TEST D'ELEMENTS D'OSMOSE INVERSE FT-30 DE 20 CM  
DE DIAMETRE AVEC EAU DE MER ET EAUX SAUMATRES

ABSTRAIT

Des éléments d'osmose inverse formés d'une membrane FT-30 ont été utilisés dans toute une série d'opérations de désalage autour du monde. La membrane a été développée d'abord pour satisfaire le besoin d'une membrane unipasse pour eau de mer qui maintiendrait ses caractéristiques de rejet de sel tout en fonctionnant avec différentes sources d'alimentation maritimes. Plus tard des résultats de test ont démontré son utilité en tant que membrane de désalage pour eaux saumâtres, et des éléments FT-30 sont en opération dans un grand nombre de conduits d'eau, de l'eau de robinet jusqu'à des séparations industrielles difficiles.

La membrane FT-30 est non-celluleuse et consiste d'une configuration composite de film mince. Elle satisfait le besoin principal de haut rendement et d'excellent rejet de sel, mais en plus elle est chimiquement stable, peut opérer sur un champ étendu de PH, résiste en partie à l'exposition à la chlorine et fait preuve de stabilité à des températures d'écoulement élevées. Ces dernières qualités facilitent le nettoyage acide et caustique, une considération importante dans des conditions d'encrassement.

Une expérience pratique considérable a été obtenue avec les éléments de la membrane FT-30 pendant les trois dernières années. Cette expérience a donné d'excellents résultats, bien que limitée à des petits éléments --le plus grand élément construit jusque là par FilmTec était de 10 X 52 cm et donnait un rendement de  $2,3 \text{ m}^3$  par jour avec de l'eau de mer et  $4,5 \text{ m}^3$  par jour dans le cas d'eaux saumâtres. Au début de 1982 des éléments de 10 X 100 cm ont commencé à être construits et utilisés dans le marché. Ensuite, en été 1982, des éléments de 20 X 100 cm ont été manufacturés pour la première fois, allant à l'encontre des buts de design pour performance unipasse avec eau de mer, soit une production d'écoulement de  $19 \text{ m}^3$  par jour et de  $38 \text{ m}^3$  par jour dans le cas d'eaux saumâtres. Ces éléments plus larges subissent en ce moment des tests avec eau de mer et eaux saumâtres au Moyen Orient, en Angleterre, en Floride, et en d'autres lieux. Les données de performance unipasse avec eau de mer et les résultats de test avec eaux saumâtres sont présentés. Des éléments de 20 cm sont maintenant en production en quantités commerciales pour servir dans des larges installations d'osmose inverse.

MESSWERTE FÜR UMGEKEHRTE OSMOSE VON MEER - UND BRACKWASSER  
MIT HILFE VON FT-30 ELEMENTEN MIT DURCHMESSER 20 CM

ZUSAMMENFASSUNG

Spiralelemente aus FT-30 Membrane sind weltweit für verschiedenartige Entsalzungsaufgaben verwendet worden. Ursprünglich wurde die Membrane entwickelt, um das Bedürfnis nach einer funktionierenden Einstufen-Meerwassermembrane zu befriedigen, welche beim Einsatz in verschiedenartigen Speisewässern aus dem Meer ihre Salzausscheidungsfähigkeit beibehalten würde. Spätere Prüfergebnisse erwiesen ihre Anwendbarkeit auch auf die Entsalzung von Brackwasser; FT-30-Elemente werden jetzt bei einer breiten Skala von Speisewässern angewendet, vom Leitungswasser bis zu komplizierten industriellen Trennungen.

Die FT-30-Membrane ist nicht zelluloseartig und besteht aus einer Dünnschichtzusammensetzung. Sie befriedigt das Hauptbedürfnis nach einer Membrane mit hohem Flussvermögen und ausgezeichneter Salzausscheidungsfähigkeit, ist aber ausserdem chemisch beständig, funktioniert in einem weiten pH-Bereich, ist teilweise chlorbeständig, und erweist sich als beständig bei hohen Speisewassertemperaturen. Die letzteren Eigenschaften ermöglichen ihre Reinigung durch Säure sowie auch durch kaustische Alkalien, was bei Verschmutzung von Bedeutung ist.

Im Laufe der letzten drei Jahre wurden beträchtliche Erfahrungen beim praktischen Einsatz von FT-30-membrane gesammelt. Die Resultate sind ausgezeichnet, doch waren sie auf kleine Elemente beschränkt -- das grösste von FilmTec hergestellte Element war bis vor kurzem 10 X 52 cm und verarbeitete 2,3 m<sup>3</sup> Meerwasser pro Tag und 4,5 m<sup>3</sup> Brackwasser pro Tag. Seit Anfang 1982 werden 10 X 100 cm-Elemente hergestellt und kommerziell verwendet. Im Sommer 1982 wurden die ersten 20 X 100 cm-Elemente hergestellt; diese Ausführung verarbeitet im Einstufenprozess pro Tag über 19 m<sup>3</sup> Meerwasser resp. über 38 m<sup>3</sup> Brackwasser. Diese grossen Elemente werden zur Zeit im Mittleren Osten, in England, Florida und an mehreren anderen Orten mit Meer- und Brackwasser erprobt. Leistungsgrössen für den Einstufenprozess für Meer- und Brackwasser werden vorgelegt. 20 cm-Elemente werden jetzt für den Gebrauch in grossen Anlagen für umgekehrte Osmose massenproduziert.

### INTRODUCTION

Since the early work of Reed and Breton (Ref. 1) about 25 years ago, followed by the important developments made by Loeb and Souriragan (Ref. 2), gradual but significant advances have been made in reverse osmosis technology. During the 1960's the major R & D effort was directed towards improving cellulose acetate membrane so that by the 1970's it had been commercially introduced into the world desalting market. Today 80 percent of reverse osmosis installations use cellulose acetate membranes.

Distillation systems, however, still make up a major portion of the world's total desalting capacity. This is due partly to its being a more mature technology, but also to the fact that cellulose acetate membranes suffer from several significant disadvantages. To a certain extent they are susceptible to biological attack, flux decline due to progressive creep and compaction phenomena at high pressures and/or temperatures, and damage at high operating pH (this also limits its cleanability). In addition, little success has been realized in obtaining high membrane fluxes, while at the same time retaining high salt rejection. In general, these limitations have resulted in more extensive system pretreatment, limited operational ranges, high pumping costs and limited operating lifetime. The end result has been unattractive capital and annual costs and poor return on investment.

New cellulose acetate membranes, such as cellulose triacetate, have higher salt rejections, but they are still bothered by low membrane flux so that their prime commercial form is as a hollow fiber (which allows for high membrane surface area per unit volume to achieve high overall throughput of water). Several companies have developed modules based on this concept. Cellulose acetate blends also have been developed commercially in recent years for spiral elements. They have improved salt rejection and greatly improved flux but are still subject to the same chemical stability limitations.

Other membranes available in hollow fiber form, such as aromatic polyamide and polyhydrazide, suffer two main disadvantages: lack of chlorine resistance and high susceptibility to fouling by colloidal materials in water. In addition, because these membranes are asymmetric in their hollow fiber form, they are thereby temperature limited (maximum recommended use temperature of 35°C).



A facility to produce 40-inch-wide membrane is now in operation, and elements up to eight inches in diameter and 40 inches long are being manufactured. The eight-inch-diameter by 40-inch-long seawater elements provide 6,500 gpd of permeate at a pressure of 800 psi. The brackish water elements in this configuration produce 6,000 gpd of permeate at a pressure of 200 psi.

#### DESCRIPTION OF FT-30 MEMBRANE

The FT-30 membrane consists of an ultrathin barrier layer about 0.2  $\mu\text{m}$  thick supported by a microporous polysulfone coating on a polyester carrier web. The barrier layer is a proprietary crosslinked aromatic polyamide having some anionic functionality. Figure 1 illustrates the structure of the membrane.

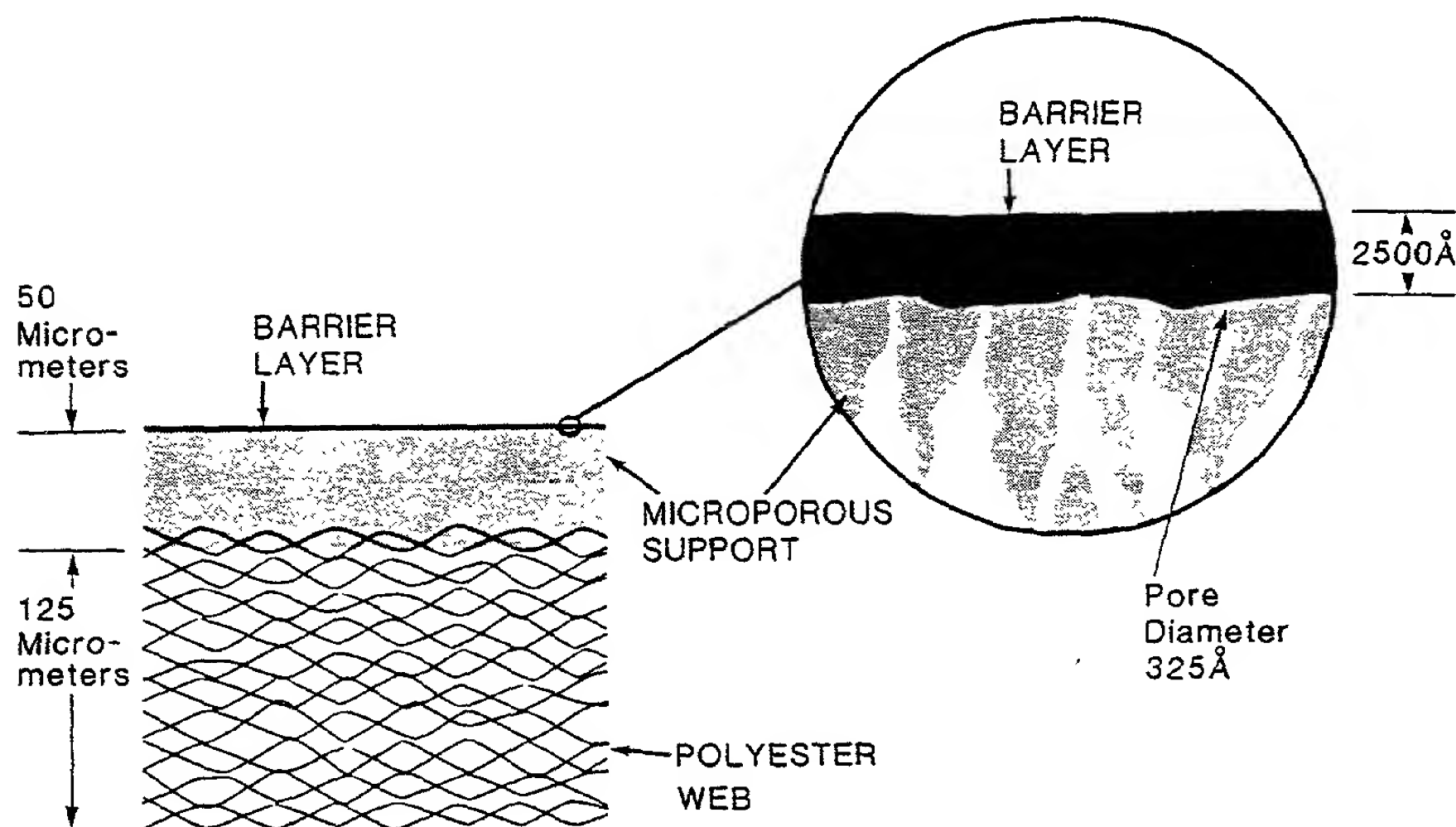


FIGURE 1. FT-30 Thin film composite membrane.

An important difference between the FT-30 membrane and other thin-film composite membrane is the thickness of the barrier layer. The FT-30 membrane has a barrier layer several times thicker than the others (about 2500 Ångstroms) which makes it much more resistant to damage by mechanical stresses and oxidizing agents. This feature, combined with its pH stability, allows the membrane to operate over a broad range of pH (3 to 11 continuously and 1 to 13 short-term) and to be cleaned with strong acid and base solutions. Another important advantage is that the membrane itself

can be exposed to boiling water temperatures without damage, although when produced in a spiral-wound configuration, lower temperature limitations must be observed. The FT-30 membrane shows considerably more resistance to chlorine and other oxidizing agents than other commercially available polyamide membranes, although it is not as good as cellulosic membranes in this regard. A complete discussion of the chemical stability of the FT-30 is presented in References 9 and 10.

#### SUMMARY OF PERFORMANCE

Basic characteristics of the FT-30 membrane may be summarized as follows. It is a high-flux, high-salt rejection membrane. Salt rejections in seawater tests are typically 99.0 to 99.2 percent for commercially produced, spiral-wound, FT-30 membrane elements. No colloidal treatment, such as tannic acid or polyvinyl methyl ether, is needed to maintain salt rejection. Salt rejections to 99.5 percent can be produced on a laboratory scale and may eventually be achieved in spiral elements. Fluxes are very good. Under seawater desalting conditions of 800 psi and 25°C, membrane fluxes of 22 gfd (gallons per square foot per day) can be routinely achieved in spiral elements. Even higher fluxes have been achieved (up to 39 gfd) in membrane manufacturing trials. A brackish water version of the membrane is also being manufactured that can provide a flux of 20 gfd at only 200 psi, giving salt rejection of 96 to 98 percent. This version offers the possibility of low-pressure brackish water desalination, with attendant reductions in equipment and operating costs. Tapwater versions of the membrane are likewise being produced that provide purified water in homes, using the tapwater line pressure as the driving force for reverse osmosis.

#### TEST RESULTS

##### Long-Term Tests

Only limited test results on eight-inch elements were available at the time of preparation of this paper and long-term tests have only just been initiated. Long-term tests on the smaller elements, which use the same membrane, are presented in this section. Test results conducted so far on the large elements indicate that they will perform in the same manner as the more extensively tested smaller units.

Brackish-water modules have been on test for over two years at the Roswell Test Facility of the U.S. Department of the Interior and at the Sanibel Island plant of the Island Water Association.

The long-term tests at Roswell are shown in Figures 2 and 3 and the Sanibel test is presented in Figure 4. In general, these tests have shown that the FT-30 membrane is capable of at least several years of operation on brackish feeds with no loss in rejection and only slight flux loss that would normally be expected with a certain amount of fouling.

Several long-term seawater tests have been carried out at the Wrightsville Beach Test Facility of the U.S. Department of Interior, by the U.S. Navy, and by the U.K. government. These results are reported in detail in References 9 and 10. In general, these tests indicate that when the effects of fouling and scaling can be minimized no loss in salt rejection and only minimal flux losses occur over long-term operation.

Long-term tests under both brackish water and seawater conditions are now in progress with the eight-inch elements at numerous locations around the world. Information on the first eleven of these tests is presented in the following table.

ELEMENT	CONFIGURATION	LOCATION	FEEDWATER
BW30-8040	6-Element Module	Orange County	Effluent
SW30-8040	6-Element Module	Mexico	Seawater
BW30-8040	1-Element Module	Florida	Brackish
BW30-8040	6-Element Module	Florida	Brackish
BW30-8040	1-Element Module	Denmark	Brackish
BW30-8040	3-Element Module	England	Seawater
BW30-8040	6-Element Module	MidEast	Brackish
SW30-8040	6-Element Module	MidEast	Seawater
SW30-8040	1-Element Module	Florida	Seawater
SW30-8040	2-Element Module	Europe	Seawater
BW30-8040	6-Element Module	Texas	Brackish

#### Preliminary Test Results with Eight-Inch Elements

The eight-inch brackish water elements have an effective membrane area of 320 square feet. The seawater version has 290 square feet. The large-element production facility produces seawater membrane that has a flux of 22 gfd and a salt rejection of about 99 percent when tested on seawater of 35,000 ppm at a pressure of 800 psi and a temperature of 25°C. The brackish water membrane has a flux of about 16 gfd and a salt rejection of about 96 percent on brackish water of 2000 ppm at a pressure of 200

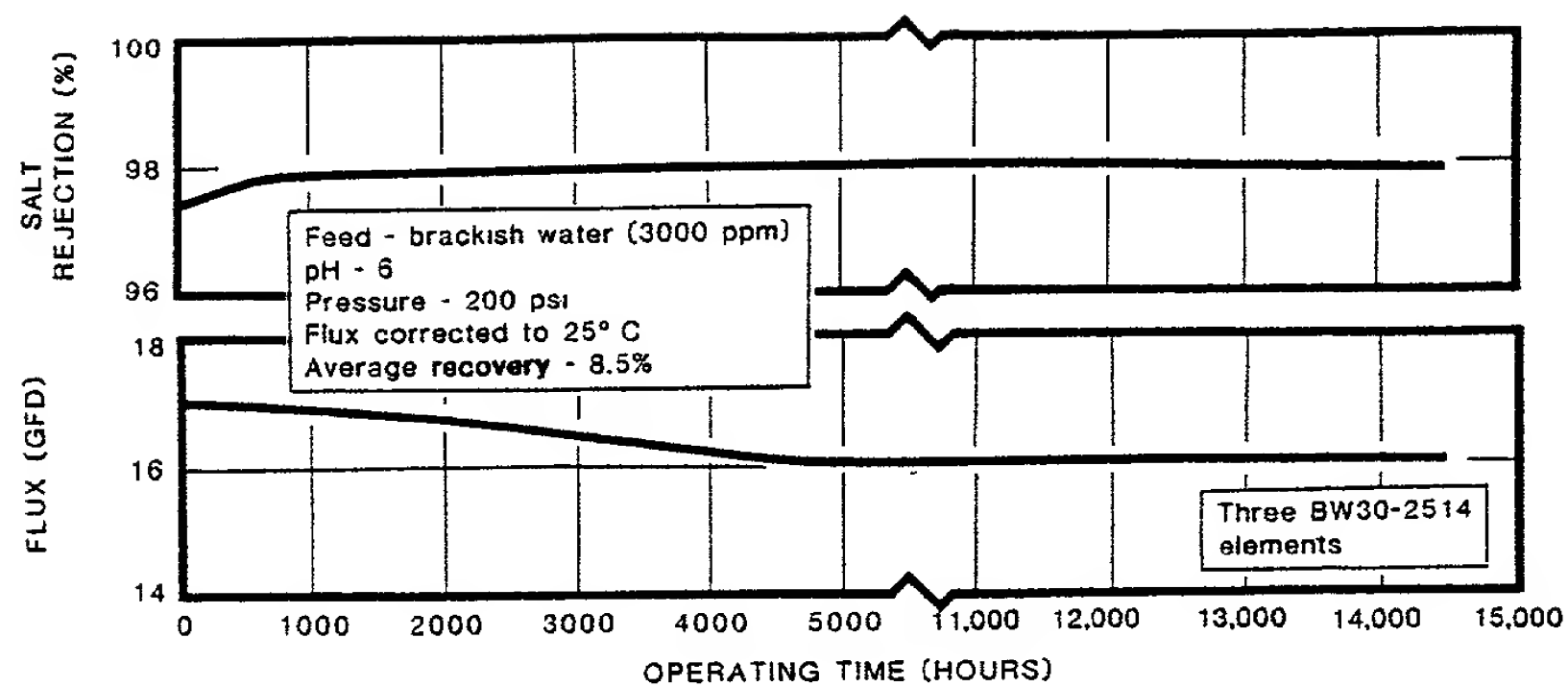


FIGURE 2. FT-30 element performance under long-term test on brackish water at Roswell Test Facility, U.S. Department of the Interior.

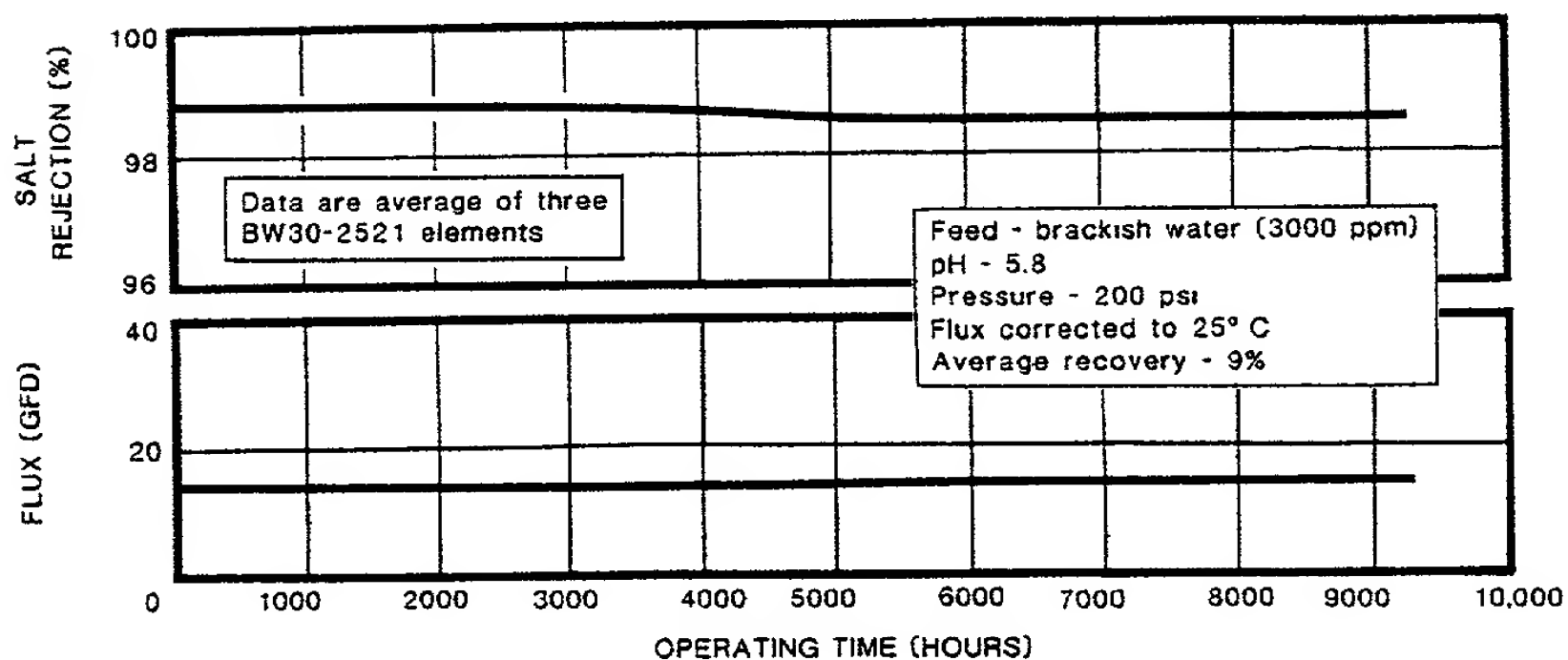


FIGURE 3. FT-30 element performance under long-term test (chlorine dioxide feed 3 ppm) on brackish water at Roswell Test Facility, U.S. Department of the Interior.

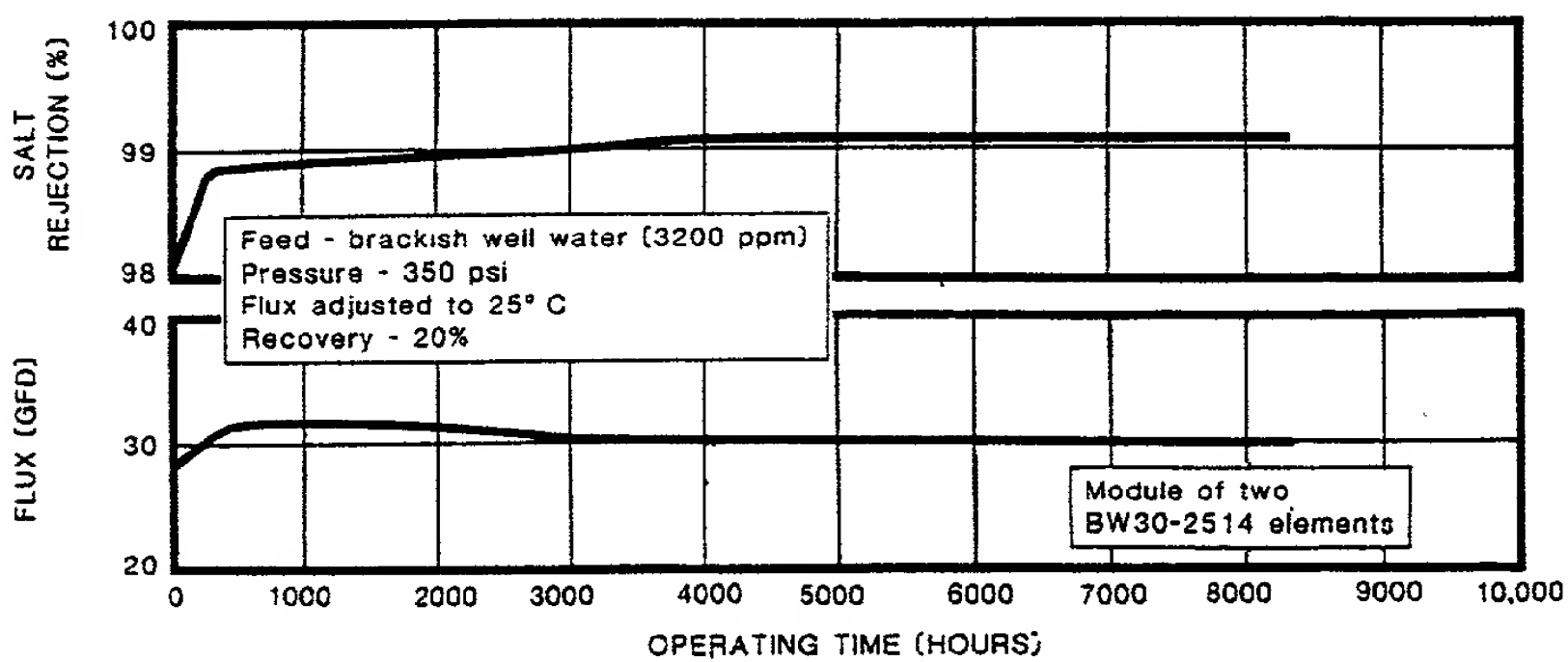


FIGURE 4. FT-30 element performance under long-term test on brackish well water at the Island Water Association Facility, Sanibel Island, Florida.



psi and a temperature of 25°C. The brackish water membrane flux will be increased to 20 gfd when the membrane machine is optimized. This is the value that has been attained on the smaller machine. These present fluxes and effective membrane areas result in element product water production capabilities of 6500 gpd and 5000 gpd (soon 6000 gpd) for the seawater and brackish water elements respectively.

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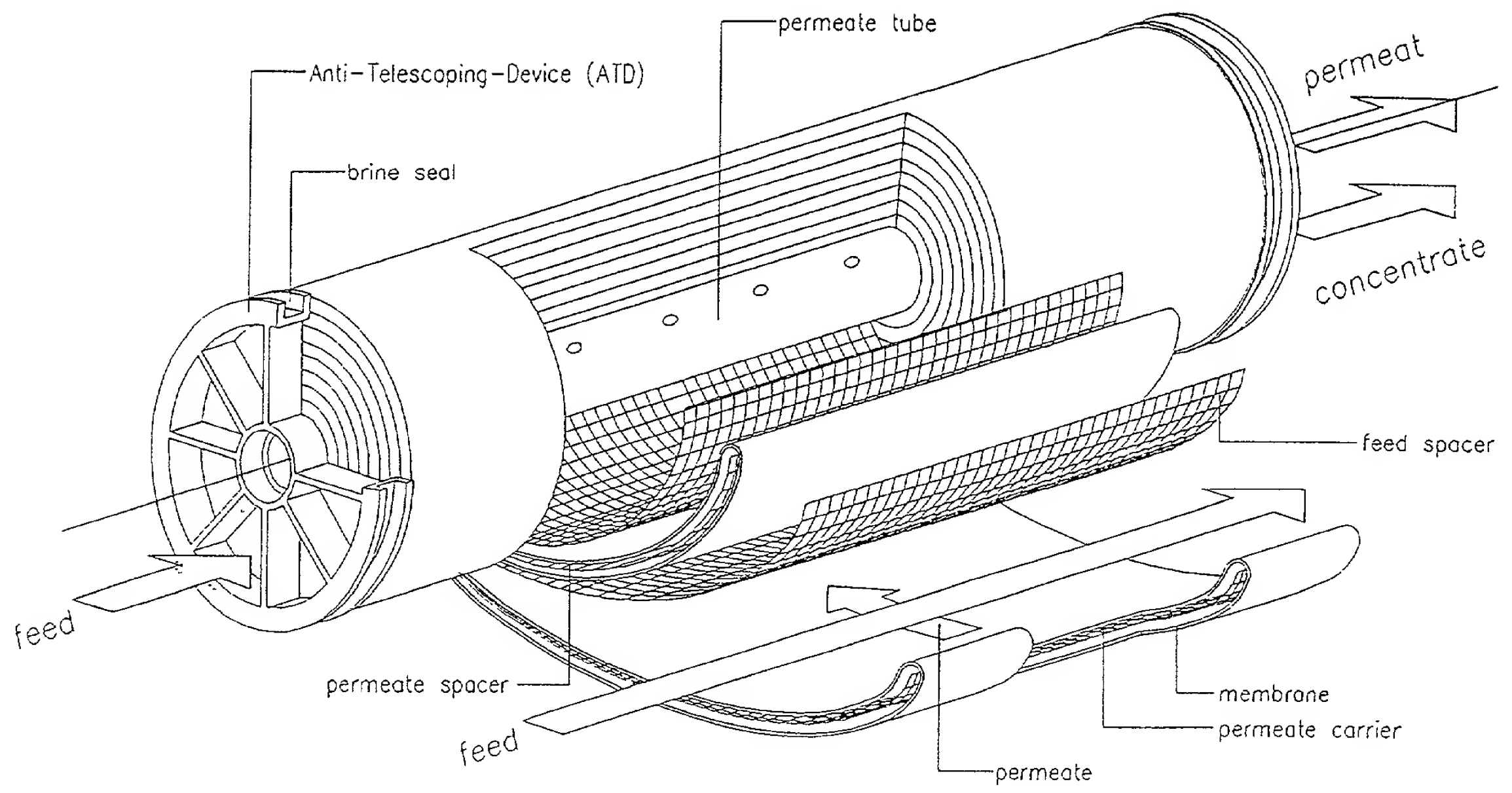


Abb. 1

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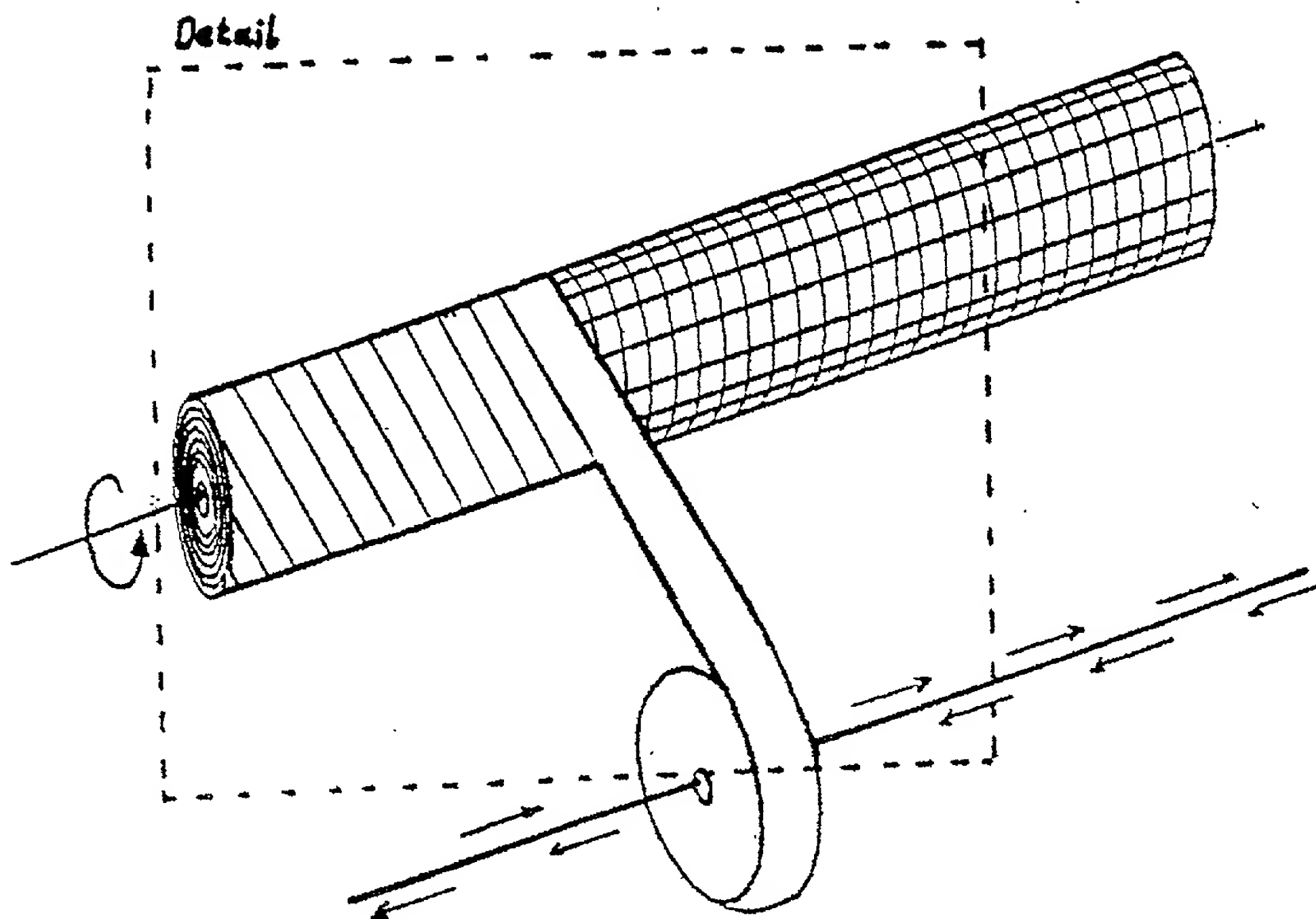


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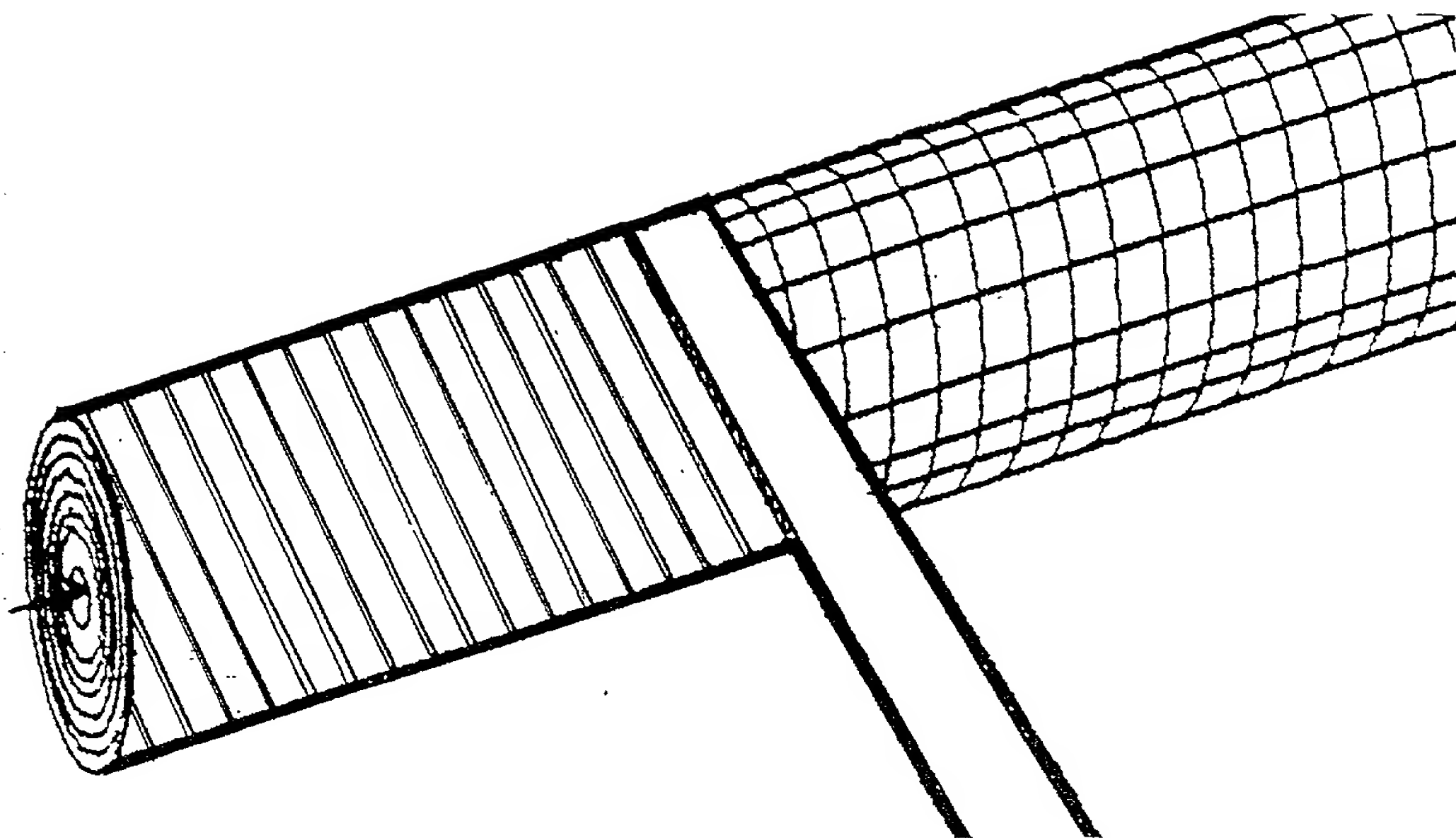


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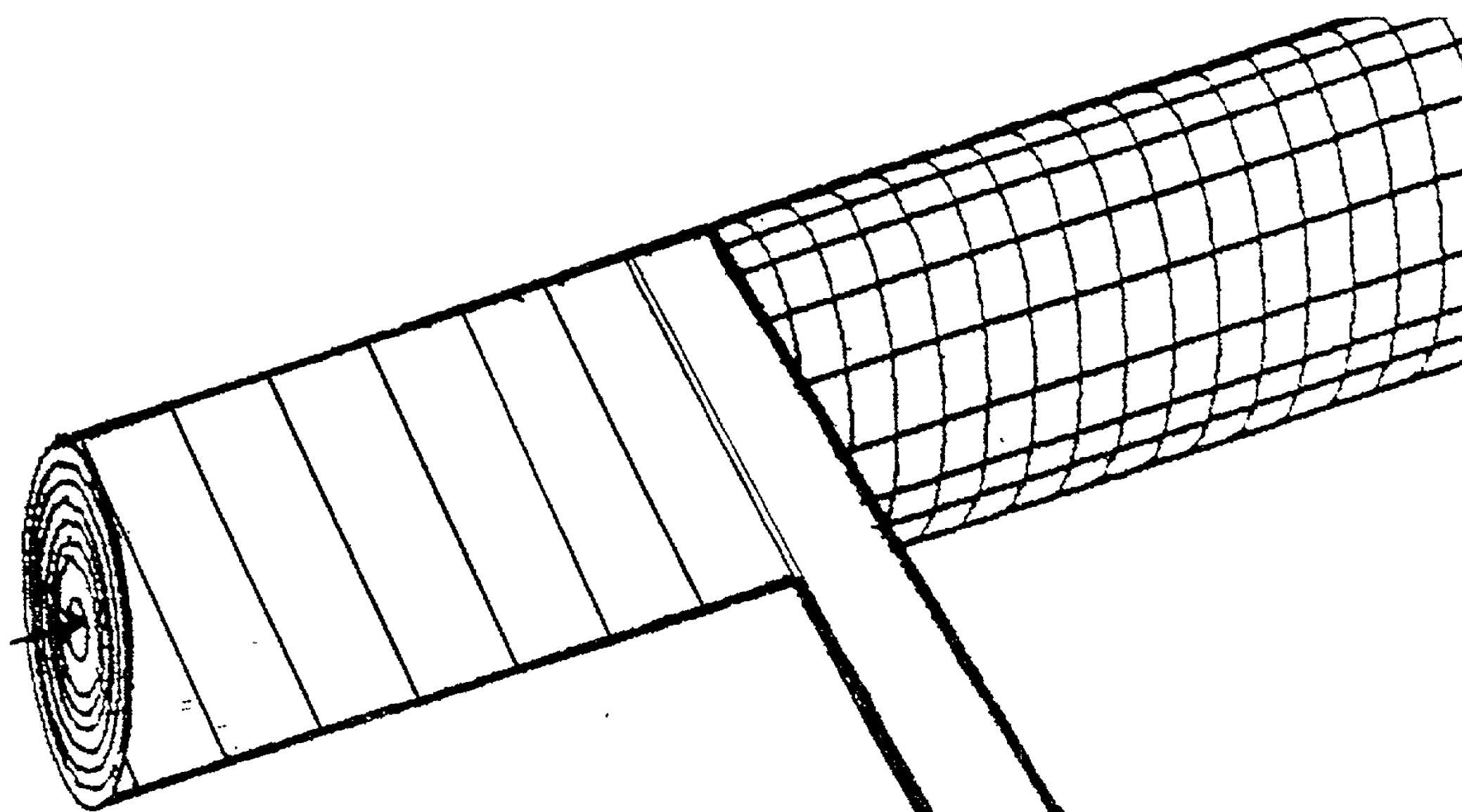


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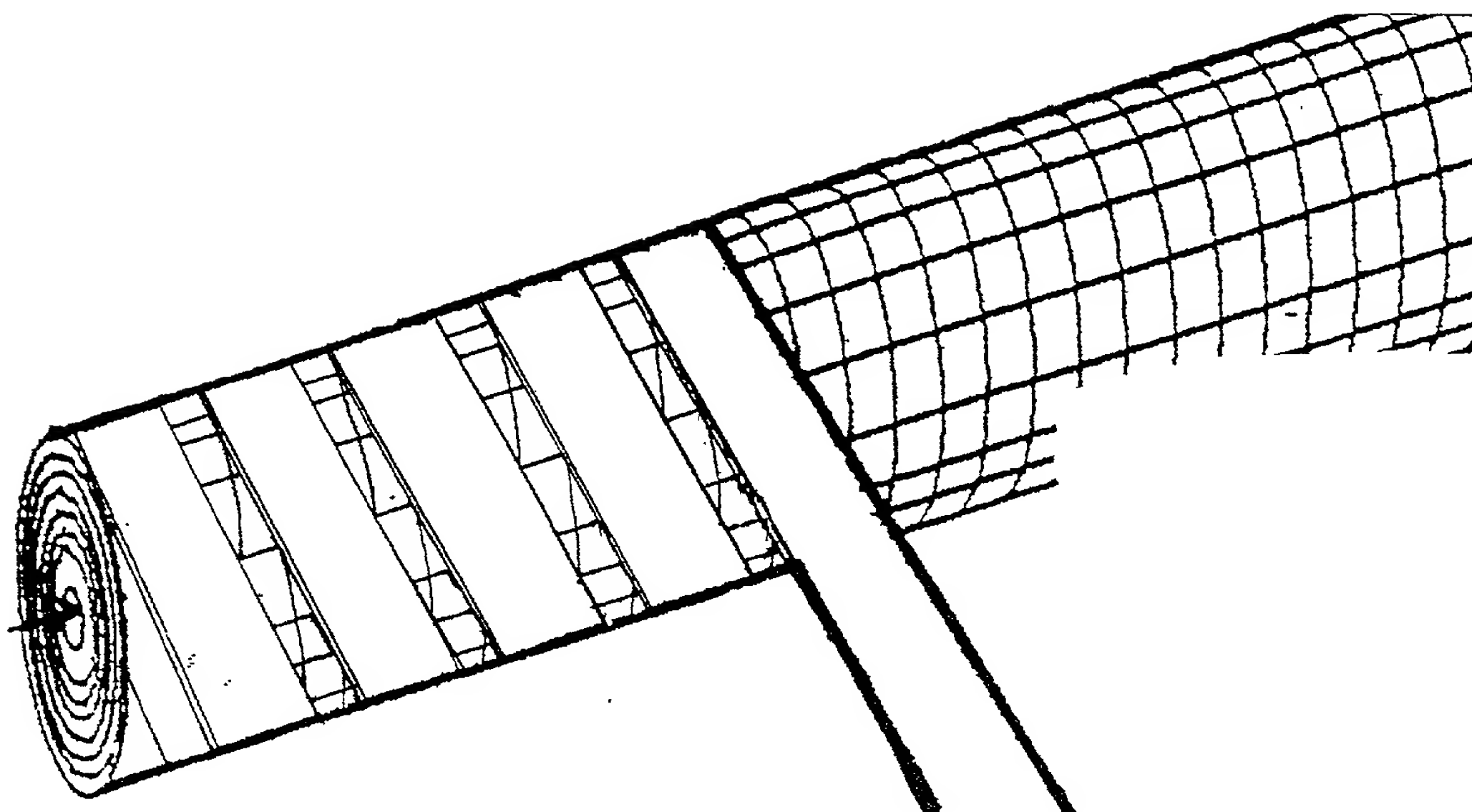


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**DECLARATION AND POWER OF ATTORNEY**

As a below-named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name.

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:

***Membrane element and process for its manufacture***

the specification of which is attached hereto unless the following box is checked:

☒ was filed on \_\_\_\_\_ as United States Application Number or PCT International Application Number \_\_\_\_\_ and was amended on \_\_\_\_\_ (if applicable).

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information which is known by me to be material to patentability as defined in Title 37, Code of Federal Regulations § 1.56.

I hereby claim foreign priority benefits under Title 35, United States Code, § 119 (a)-(d) or § 365 (b) of any foreign application(s) for patent or inventor's certificate, or § 365 (a) of any PCT International Application which designated at least one country other than the United States, listed below and have also identified below any foreign application for patent or inventor's certificate, or PCT International application having a filing date before that of the application on which priority is claimed:

**PRIOR FOREIGN APPLICATION(S)**

NUMBER	COUNTRY	DAY/MONTH/YEAR FILED	PRIORITY CLAIMED
198 26 161.6	Federal Republic of Germany	June 12, 1998	Yes

I hereby claim the benefit under Title 35, United States Code §119 (e) of any United States provisional application(s) listed below:

APPLICATION NUMBER	FILING DATE

I hereby claim the benefit under Title 35, United States Code, § 120 of any United States application(s), or § 365 (c) of any PCT International application designating the United States, listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States or PCT International application in the manner provided by the first paragraph of Title 35, United States Code, § 112, I acknowledge the duty to disclose information which is known by me to be material to patentability as defined in Title 37, Code of Federal Regulations § 1.56 which became available between the filing date of the prior application and the national or PCT International filing date of this application:

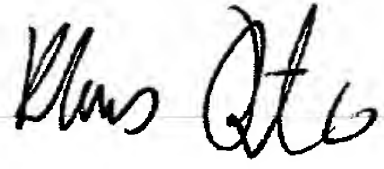
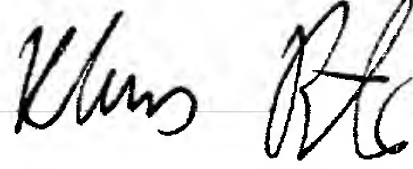
APPLICATION SERIAL NO.	FILING DATE	STATUS: PATENTED, PENDING, ABANDONED

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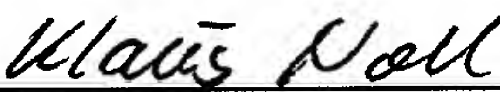
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I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of this application or any patent issued thereon.

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